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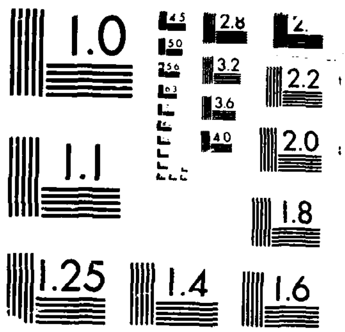

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EFFECTS OF APPLIED STRESS AND TEMPERATURE ON THE NONLINEAR ELASTIC PROPERTIES OF GRAPHITE FIBERS

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FOR NAVAL SURFACE WARFARE CENTER
RESEARCH AND TECHNOLOGY DEPARTMENT

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The nonlinear elastic behavior of a series of pitch-based graphite fiber bundles was determined by using a new measurement method that uses laser-generated ultrasound over ranges of temperature and static tensile stress. From these data, plots are presented of the Young's modulus versus stress and temperature. The modulus increases with static tensile stress and decreases with temperature. These results are important for characterizing the elastic behavior of composites in which these fibers are used. Some possible mechanisms for explaining the observed effects are discussed.

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FOREWORD

This report presents data for the dependence on applied stress and temperature of pitch-based graphite fiber bundles, obtained through a novel technique of laser-generated ultrasound. Such data are indispensable for describing the effects of temperature on composites reinforced by these fibers and should assist in the selection of constituents for making composites of high thermal stability.

This work was performed during the period of September 1986 to February 1987. The technical program monitor is Dr. John M. Liu.

Approved by:

James F. Goff (act)

James F. Goff, Acting Head
Materials Division

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INTRODUCTION

The existence of carbon fibers has been known for many years. They were used for filaments for incandescent lamps in the latter part of the 19th century. The first major effort to develop high strength carbon fibers, however, was not undertaken until the early 1950's at Wright Patterson Air Force Base, Dayton, Ohio.[1] Such fibers are now important in many advanced technical applications, especially in aerospace composite materials for which the high specific modulus is valuable. Care must be taken in the design of these composites, as fiber and matrix will have significantly different properties. The fibers exhibit nonlinear elasticity,[2] as well as a negative coefficient of thermal expansion parallel to the fiber axis.[3] These factors can result in important changes in the properties of a composite subjected to wide variations in temperature. For example, the Young's modulus along the fiber direction in a uniaxial lamina can increase rather than decrease with rising temperature.[4] The stresses that are generated in the composite[5] can lead to yielding at the fiber-matrix interface, or possibly in the matrix or the fibers themselves. Furthermore, these effects will lead to variations in both the sign and magnitude of the coefficient of thermal expansion in the fiber direction, depending on the thermal history of the composite.[6]

There are two recently published studies on the nonlinear elasticity of pitch-based[7] and PAN-based[8] graphite fibers. In Reference 8, the results for a single filament and an impregnated tow have been used for interpreting the nonlinear behavior in composites reinforced by these fibers.

A thorough understanding of the nonlinear elastic properties of the fibers, as well as the thermal effects, is vital to the design of composites that will be stable over a wide temperature range. The considerations above are important for structures ranging in size from space stations and aircraft to circuit boards. Nevertheless, there has been no systematic investigation of these effects. This report summarizes the results of research on a series of pitch-based carbon fibers by laser-generated ultrasound. The Young's modulus is determined as a function of applied static tensile stress over a wide range of temperatures for the fibers. The goal is to provide data on the nonlinear elastic property of the graphite fibers and its temperature dependence, as well as to explain these effects in terms of the internal structure of the fibers.

EXPERIMENTAL PROCEDURES

The experimental arrangement is shown in Figure 1. A pulsed laser is used to generate stress waves in the fiber bundle by a rapid deposition of energy in a manner similar to that reported previously for fibers at room temperature without static stress.[9] A Nd-Yag laser is used to produce single pulses of about 15 ns duration and about 20 mJ of energy. The light (532 nm) is focused on the fiber, which is enclosed in a temperature cell. The fiber is mounted vertically, perpendicular to the direction of the laser beam. The ends of the fiber bundle are sandwiched between cardboard squares held together by epoxy. It is held at one end by a clamp. A hole is punched in the bottom square to allow tensile stress to be applied by the addition of weight there.

The stress is determined by dividing force by the cross-sectional area. Force is calculated from the mass loaded onto the fiber bundle. The cross-sectional area is found by dividing the mass, M, of a bundle of fibers of length, l, by the density and the length. A piezoelectric transducer is clamped near the top of the bundle to detect the acoustic wave generated by the interaction of the laser light with the bundle. (Typically, 2000 fiber bundles were used.) A sampling oscilloscope is triggered when the laser is fired by the signal from a photodiode placed near the bundle and facing the beam. The signal from the transducer is amplified, filtered, and recorded at either a 20- or a 50-ns sampling period. From this, a time of flight may be recorded as the difference in time between the triggering of the photodiode and the arrival of the acoustic wave at the transducer. A differential measurement is taken by dividing the difference between two distances from laser impact to transducer by the difference in the corresponding times of flight (i.e., velocity, $C = \Delta l/\Delta t$). As the ultrasonic wavelength is very large compared to the diameter of the bundle, Young's modulus, E, is equal to the density of the fibers times the square of the ultrasonic velocity. The differential measurement will reduce the error from both end effects and temperature gradients.

Some simple steps must be taken to ensure accurate results. The fibers must be long enough to eliminate interference in the signal from end reflections. The fibers must be straight, unbroken, and of equal length. The standard deviation for measurements of the modulus is about 3 GPa. This number is comparable to the limit of resolution, which varies a little depending on the sampling rate used and the modulus of the fiber. Over the length of sample used, l, the maximum temperature differences were $+5^{\circ}C$ at $285^{\circ}C$, less than $1^{\circ}C$ at $60^{\circ}C$, and $0^{\circ}C$ at room temperature. The fibers were pitch-based P25, P55, P75, P100, and P120 that were manufactured by the Union Carbide Corporation.

RESULTS AND DISCUSSION

Table 1 lists the results obtained at ambient temperature by both the ultrasonic method and by conventional mechanical testing. Within the limits of error for the two techniques, the results are in agreement. Interestingly, the ultrasonic velocity measured for P120, 19.5 km s^{-1}, is greater than that reported for diamond.[10]

Figures 2-6 show the values of Young's modulus plotted as a function of static tensile stress at 25, 60, 120, 210 and 285°C, respectively. The data points are averages of several measurements. All the data are reversible for stress and temperature. In general, the nonlinear elasticity decreases with increasing initial modulus in the series from P25 to P120. As a result, the fractional change in the initial modulus E_o, with respect to stress, evaluated at a stress of 10 MPa for each of the fibers, decreases with increasing initial modulus (as shown in Figure 7). This quantity is one of those that determine the unusual increase with temperature of the axial Young's modulus of a uniaxial lamina.[4]

When the temperature is increased, the modulus decreases as shown in Figure 8 for zero tensile stress and in Figure 9 for 100 MPa tensile stress. The rate of this decrease is larger for all the fibers at temperatures between about 30 and 150°C rather than at the higher temperatures. The fractional change in the initial modulus with respect to temperature decreases with increasing initial modulus (as shown in Figure 10). This temperature derivative exhibits a dependence on the initial modulus of the fibers that is similar to that exhibited by the stress derivative (as shown previously in Figure 7). This derivative is another of the quantities that determine the unusual thermal behavior of uniaxial laminae.[4] The stress derivative of each of the fibers is a function of temperature. The derivatives at room temperature and at 285°C are significantly different for the fibers of low modulus, but this difference diminishes progressively as the Young's modulus of the fiber increases, as shown in Figure 11.

In general, the initial modulus in the series of fibers studied can be associated with the extent of alignment of the closed-pack planes in graphite towards the fiber axis.[11] Crystal size in the direction of the fiber axis might also play a role. Change in crystal orientation with static tensile stress no doubt contributes to the nonlinear elasticity.[2] However, this idea has not been examined systematically for a series of fibers such as the present ones. The sharp increase in modulus with stress, such as that observed at 30°C for P25 in Figure 2, has been attributed to the motion of dislocations in the basal plane.[2] The large decrease in the modulus of P55 between 60 and 120°C (Figure 8) together with the change in the nature of the curve for this fiber between 60 and 120°C is consistent with the dislocation hypothesis, as are the

changes in the initial modulus versus temperature shown in Figure 8. The greater slopes of the curves in the 30 to 150°C range are consistent with the effect of dislocation motion on a modulus determined ultrasonically.[12] However, this hypothesis too has not been examined for a series of fibers such as the present ones. In fact, it has been suggested that the initial portion of a curve like that for P25 in Figure 2 or P55 in Figure 4 results from the fibers not being straight.[13] To address such questions, X-ray diffraction measurements will be used to determine orientation, size, and crystal modulus in the axial direction, and if possible, the degree of imperfection in the crystals. The ultrasonic measurements of Young's modulus will also be extended to higher and lower temperatures.

CONCLUSION

A new method has been developed to measure Young's modulus for graphite and other fibers over wide ranges of temperature and static tensile stress. The method is based on laser-generated ultrasound. It was used to measure the Young's modulus of pitch-based graphite fibers (P25, P55, P75, P100, and P120) from 25°C to 285°C with applied static tensile stresses from zero to about 170 MPa. The fibers exhibit nonlinear elasticity which varies in character with the fiber, the temperature, and the applied stress. The nonlinear elasticity combined with the negative axial expansion coefficient of the fibers could lead to some unusual properties in uniaxial laminae, and must be considered in the design of composites. Further sonic measurements over a wider range of temperatures and X-ray diffraction measurements are planned in order to determine the origin of the effects.

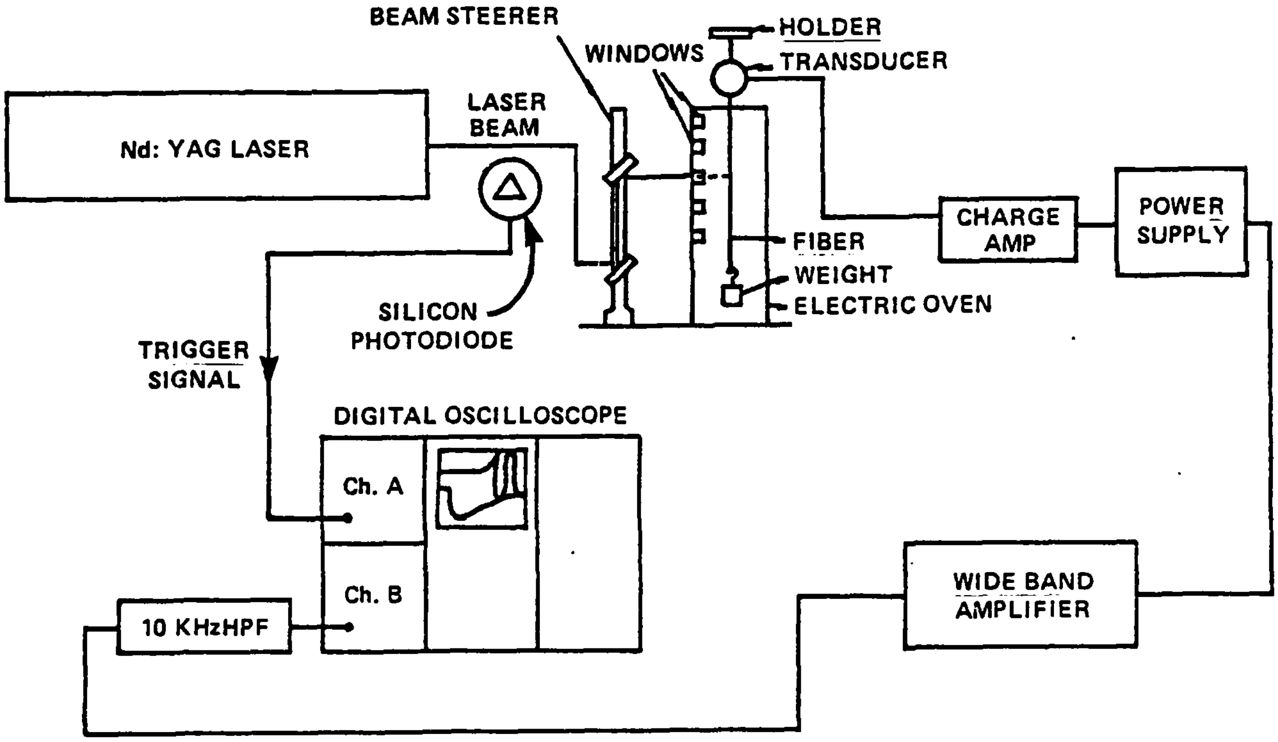

FIGURE 1. SCHEMATIC DIAGRAM OF EXPERIMENTAL SETUP FOR THE MEASUREMENT OF DYNAMIC YOUNG'S MODULUS OF A FIBER BUNDLE BY LASER-GENERATED ULTRASOUND

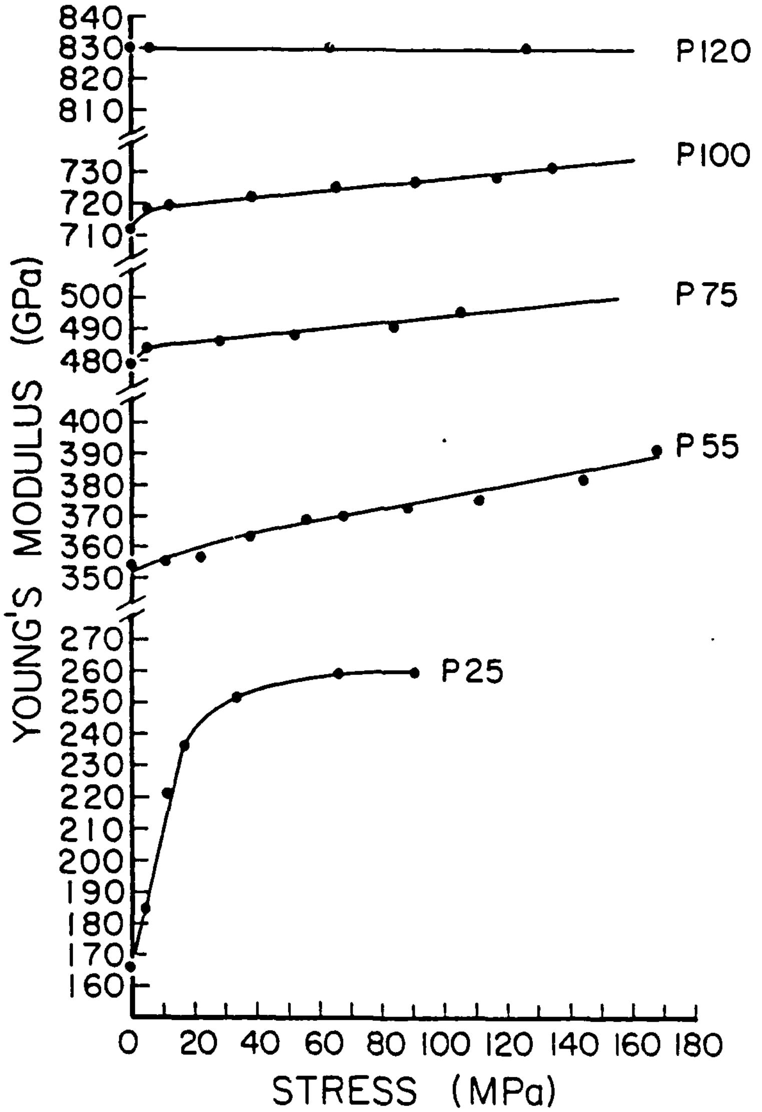

FIGURE 2. STRESS DEPENDENCE OF THE YOUNG'S MODULUS AT 25°C

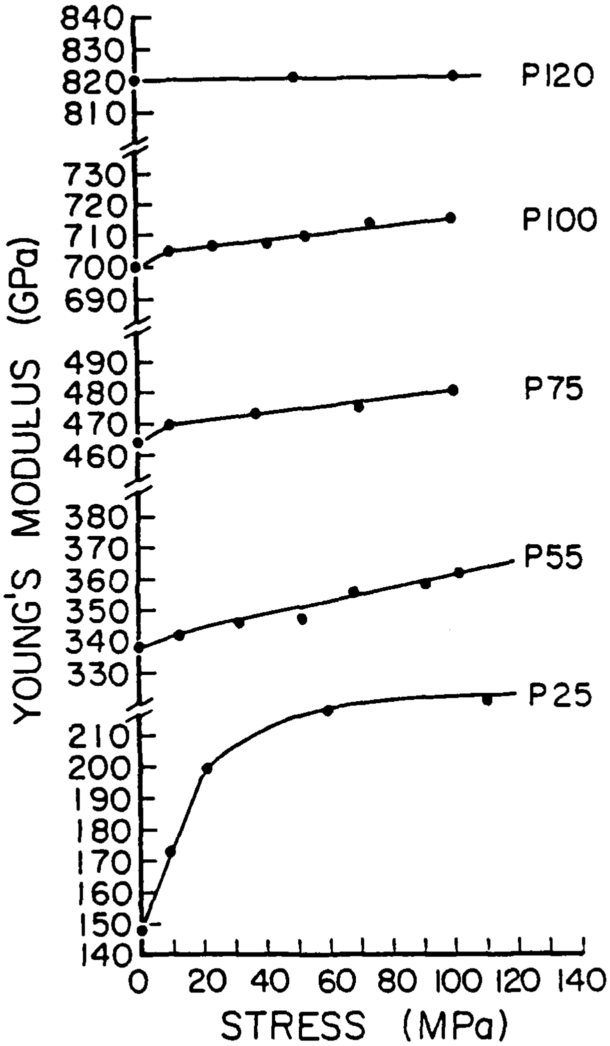

FIGURE 3. STRESS DEPENDENCE OF THE YOUNG'S MODULUS AT 60°C

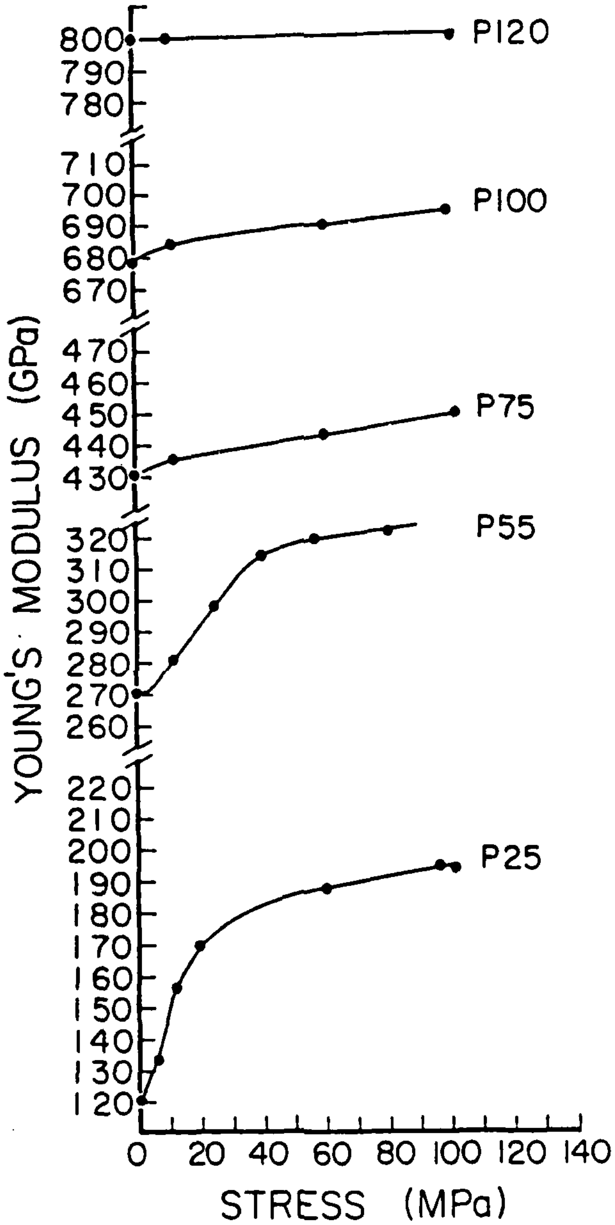

FIGURE 4. STRESS DEPENDENCE OF THE YOUNG'S MODULUS AT 120°C

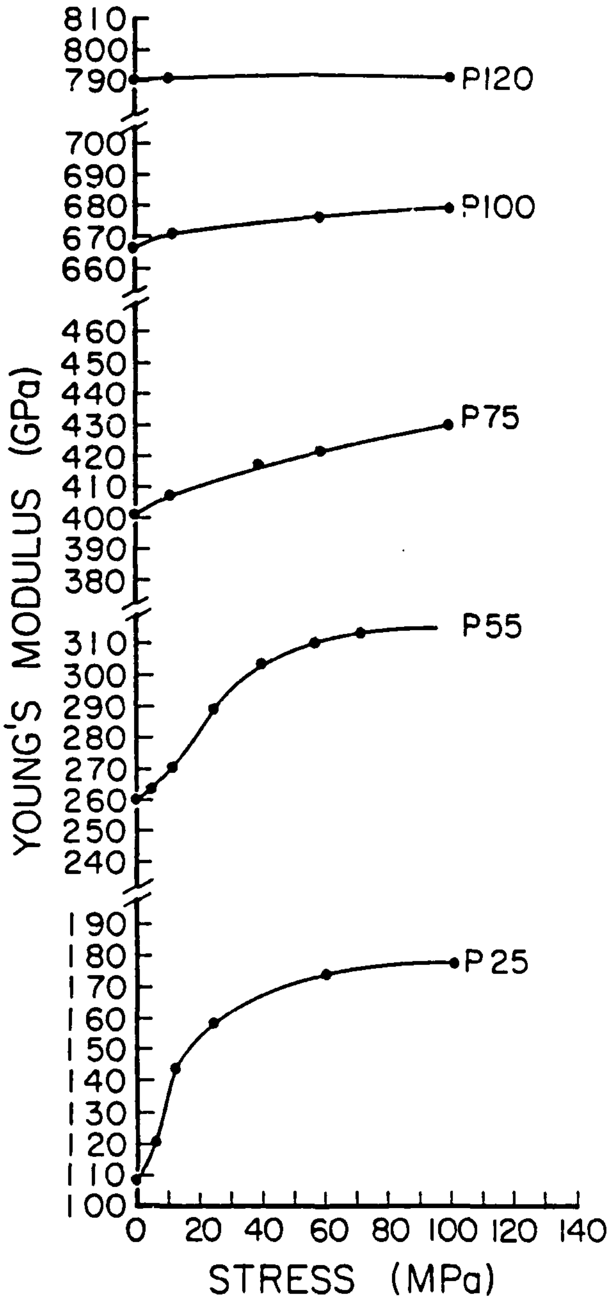

FIGURE 5. STRESS DEPENDENCE OF THE YOUNG'S MODULUS AT 210°C

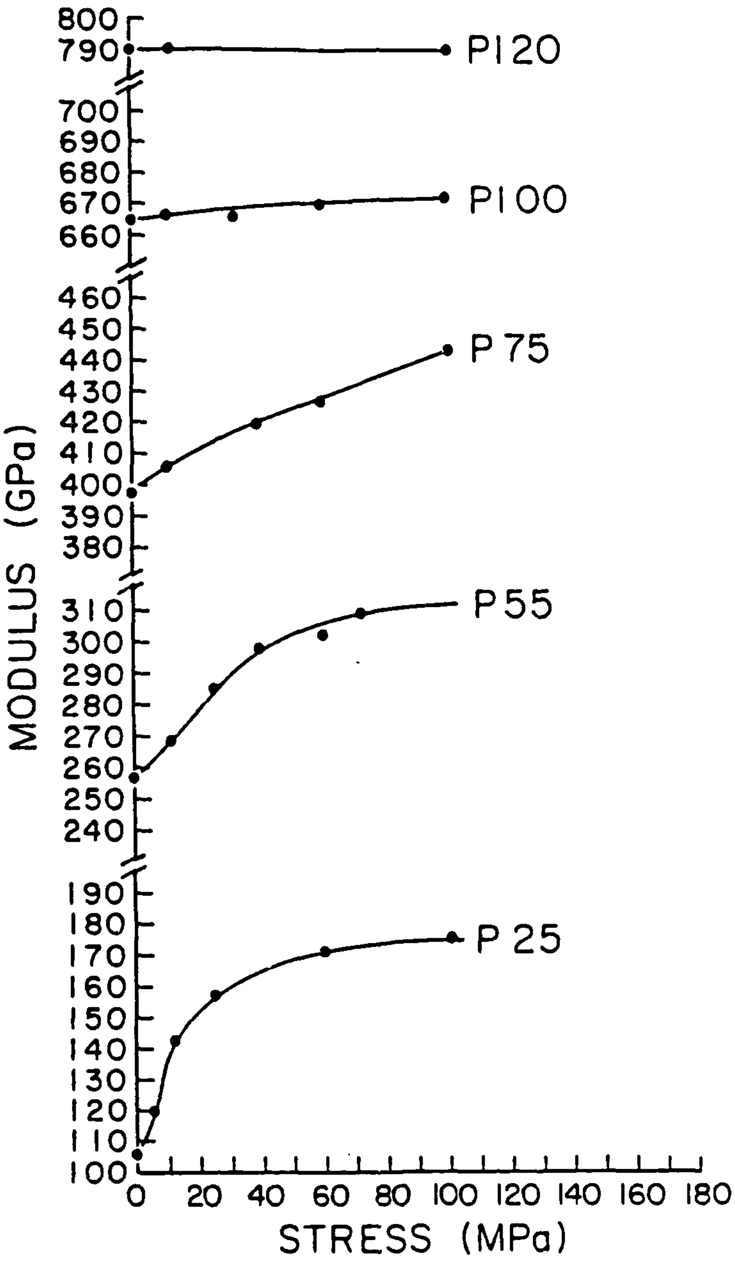

FIGURE 6. STRESS DEPENDENCE OF THE YOUNG'S MODULUS AT 285°C

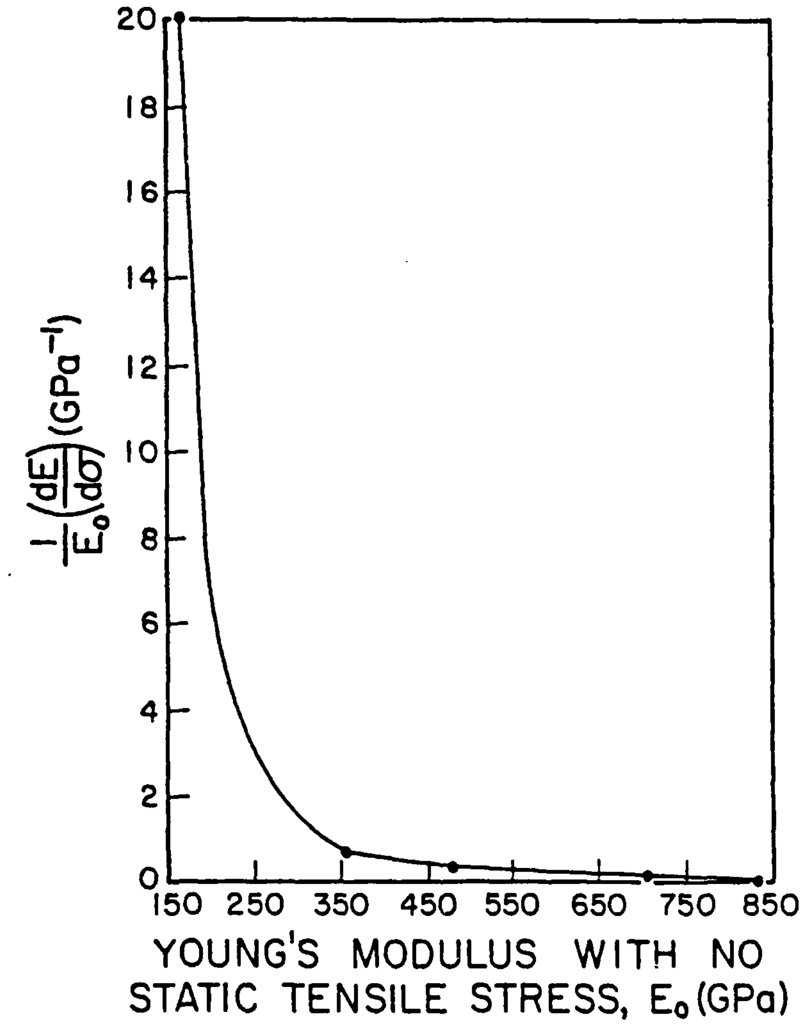

FIGURE 7. DEPENDENCE OF THE STRESS DERIVATIVE AT 10 MPa OF THE INITIAL YOUNG'S MODULUS ON THE VALUE OF THIS MODULUS

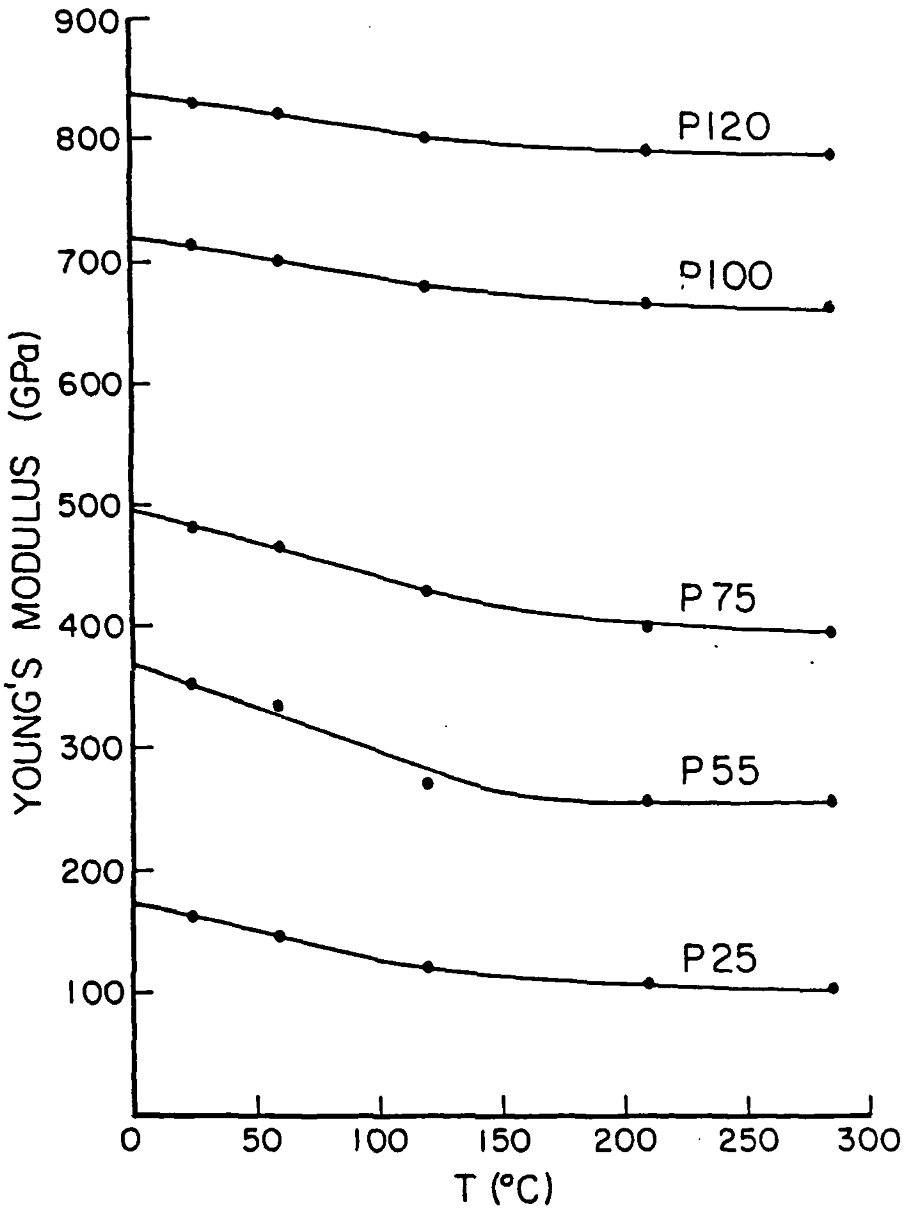

FIGURE 8. DEPENDENCE OF THE YOUNG'S MODULUS OF PITCH-BASED GRAPHITE FIBERS ON TEMPERATURE AT ZERO APPLIED STRESS

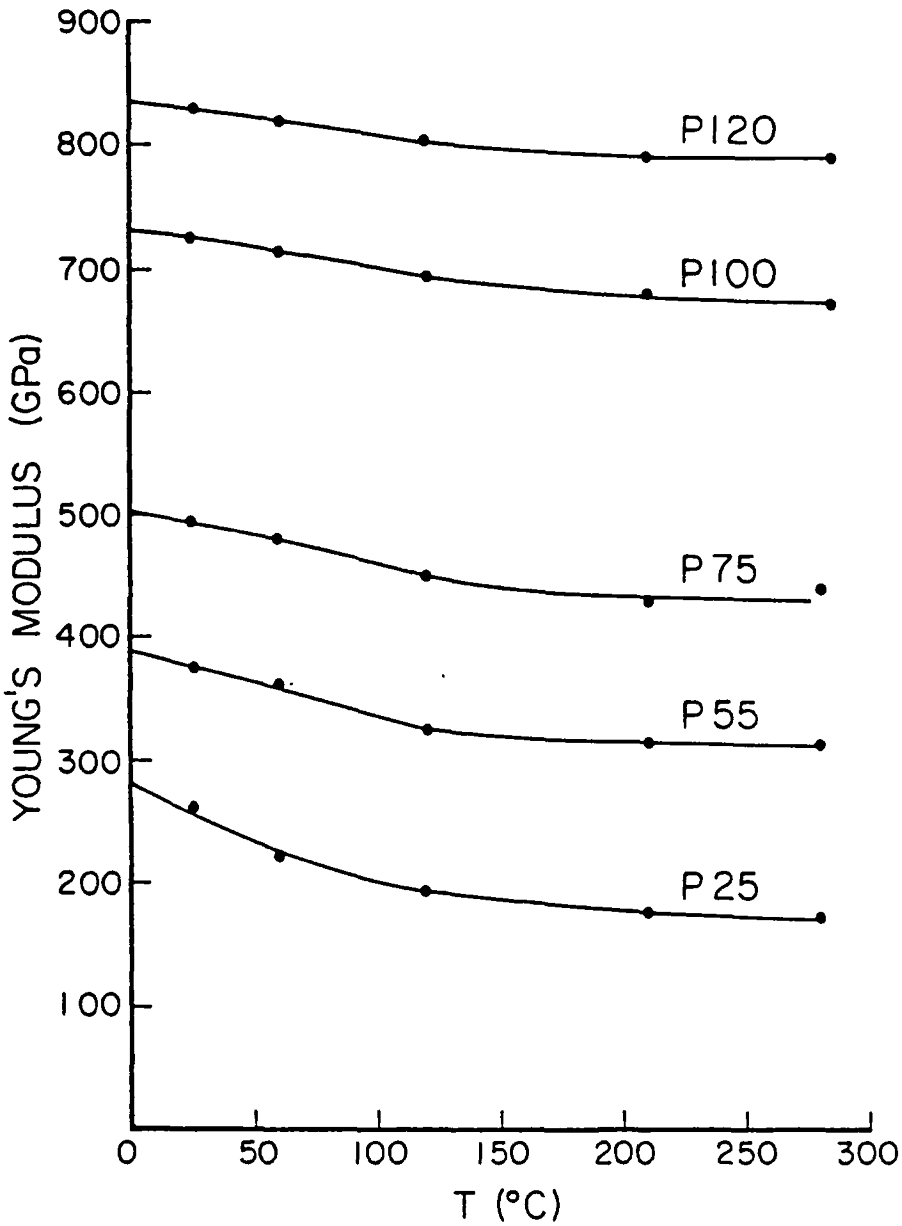

FIGURE 9. DEPENDENCE OF THE YOUNG'S MODULUS OF PITCH-BASED GRAPHITE FIBERS ON TEMPERATURE AT AN APPLIED STRESS OF 100 MPa

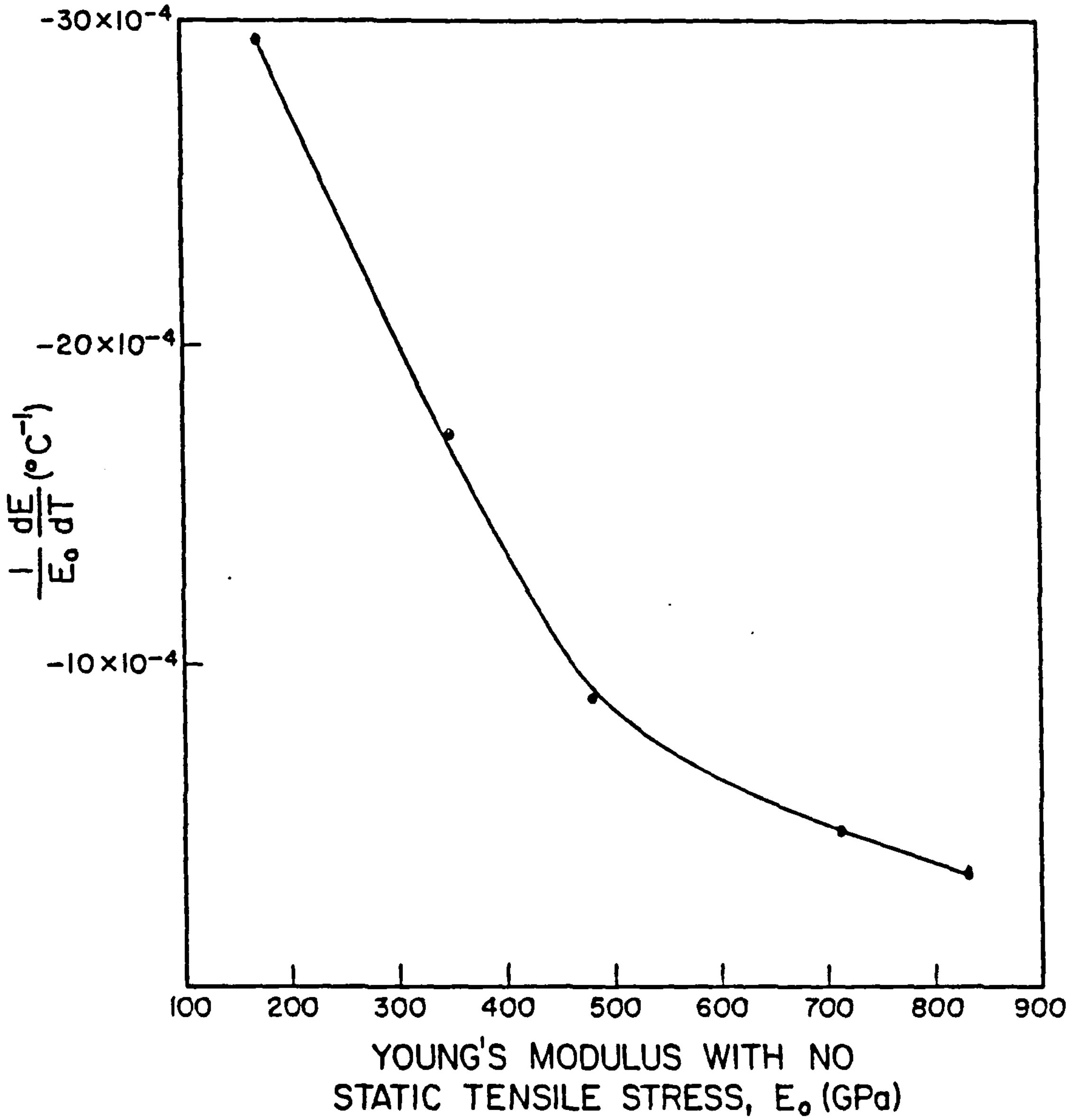

FIGURE 10. DEPENDENCE OF THE TEMPERATURE DERIVATIVE OF THE INITIAL YOUNG'S MODULUS ON THE VALUE OF THIS MODULUS

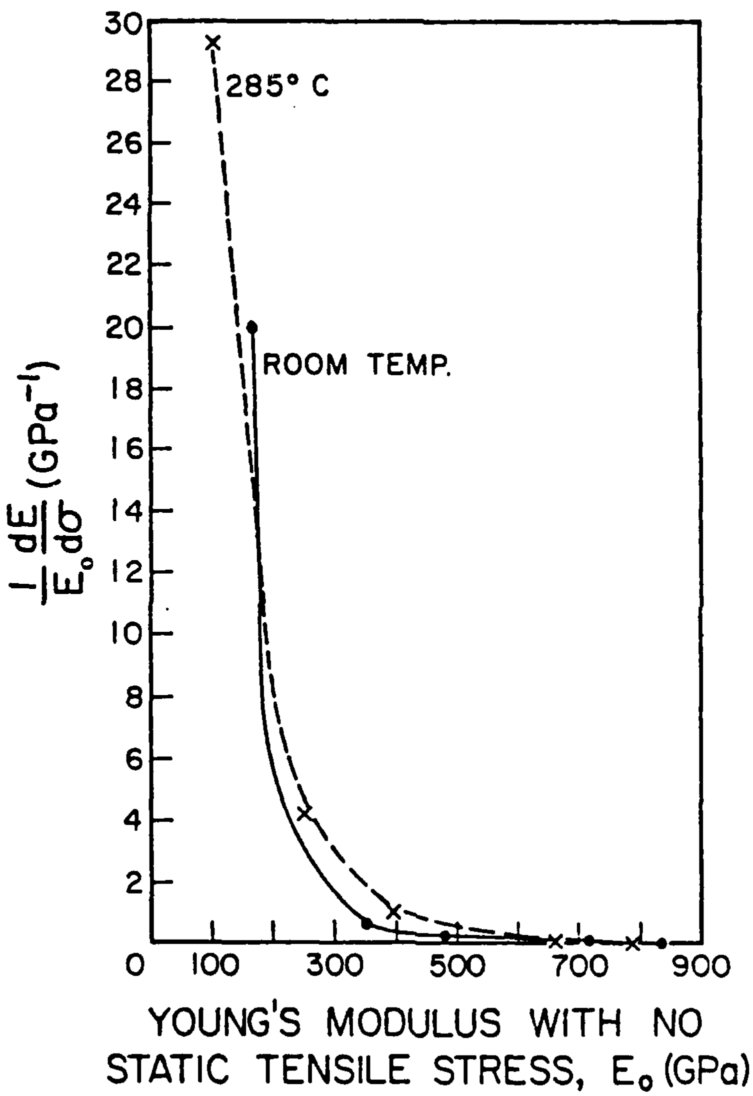

FIGURE 11. DEPENDENCE OF THE STRESS DERIVATIVES ON THE VALUES OF THE YOUNG'S MODULUS WITHOUT APPLIED STRESS AT 25°C (SOLID CURVE) AND AT 285°C (DASHED CURVE)

TABLE 1. YOUNG'S MODULUS OF PITCH-BASED GRAPHITE FIBERS DETERMINED BY LASER-GENERATED ULTRASOUND AND BY MECHANICAL TESTING

Sample	Density (Mg m^{-3})	Approximate Frequency (MHz)	Ultrasonic Velocity (km s^{-1})	Ultrasonic Modulus (GPa)	Test Machine Modulus (GPa)
P-25	1.90*	0.58	9.36	166	160*
P-55	2.00*	0.61	13.3	354	380*
P-75	2.00*	0.51	15.5	480	520*
P-100	2.15*	0.65	18.2	712	724*
P-120	2.18*	0.66	19.5	830	827*

*Values from manufacturer's data sheet

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